

Claims

- [c1] 1. A method of increasing the sequential rate at which of microwave assisted chemical reactions that potentially generate high pressure can be carried out, the method comprising:
- opening a pressure-resistant valve on a microwave-transparent pressure-resistant vessel to define a unpressurized pathway through the valve into the vessel;
 - inserting a tube through the pathway in the valve and into the vessel;
 - transferring at least one composition into the vessel through the tube;
 - removing the tube from the vessel and from the pathway in the valve;
 - closing the valve to seal the vessel against pressure release; and
 - exposing the vessel and its contents to microwave radiation.
- [c2] 2. A method according to Claim 1 comprising:
- reopening the valve after the exposure to microwave radiation to vent any high pressure from the vessel;
 - thereafter inserting the tube through the valve into the

vessel; and

thereafter removing contents from the vessel through the tube.

[c3] 3. A method according to Claim 2 comprising venting the vessel without opening the pathway for the tube.

[c4] 4. A method according to Claim 2 comprising:
adding a new composition through the tube and into the vessel following the step of removing the contents from the vessel; and
thereafter repeating the steps of removing the tube, closing the valve, and exposing the vessel and contents to microwave radiation.

[c5] 5. A method according to Claim 4 further comprising:
adding a rinsing composition to the vessel through the tube; and
removing the rinsing composition from the vessel through the tube;
both between the steps of removing contents from the vessel and adding the new composition to the vessel.

[c6] 6. A method according to Claim 1 comprising monitoring the temperature of the vessel and its contents and moderating the application of microwave energy based upon the monitored temperature.

- [c7] 7. A method according to Claim 1 comprising monitoring the pressure inside the vessel and moderating the application of microwave energy based upon the monitored pressure.
- [c8] 8. A method according to Claim 1 wherein the step of transferring the composition into the vessel comprises transferring a composition from a source library.
- [c9] 9. A method according to Claim 2 wherein the step of removing the contents from the vessel comprises forwarding the removed contents to a product library.
- [c10] 10. A vessel assembly for increasing the rate at which high-pressure sequential microwave-assisted reactions can be carried out, said assembly comprising:
a pressure-resistant reaction vessel formed of a material that is substantially transparent to microwave radiation for holding compositions that can be exposed to microwave radiation while in said vessel;
a pressure-retaining sealing valve at the mouth of said vessel that can be alternately oriented to include at least one open pathway entirely through said valve into said vessel and to seal said vessel against pressure release;
a tube for extending through said open pathway in said valve and into said vessel for providing composition

communication with said vessel through said tube;
means for mechanically inserting and retracting said tube into and from said vessel through said pathway in said valve so that compositions can be added to said vessel when said valve is open and said tube is inserted and so that said valve can be closed to seal said vessel to retain pressure therein when said tube is retracted.

[c11] 11. A vessel assembly according to Claim 10 wherein said valve can additionally be oriented to vent said vessel but without opening said pathway for said tube.

[c12] 12. A vessel assembly according to Claim 10 wherein said tube is substantially resistant to acids, bases, organic solvents and organic compositions.

[c13] 13. A vessel assembly according to Claim 12 wherein said tube comprises a fluorinated polymer.

[c14] 14. A vessel assembly according to Claim 10 wherein said reaction vessel is formed of a material selected from the group consisting of glass, quartz and polymers.

[c15] 15. An instrument for increasing the rate at which high-pressure sequential microwave-assisted reactions can be carried out, said instrument comprising:
a source of microwave radiation;
a cavity in microwave communication with said source;

an attenuator that forms at least a portion of said cavity;
a pressure-resistant microwave-transparent reaction vessel having portions in said cavity and portions in said attenuator;

a pressure-resistant valve on the mouth of said vessel;

a reciprocating tube for passing through said valve and into said vessel when said valve is open;

means for mechanically inserting and retracting the tube through said valve and into said vessel when said vessel is in said cavity and said attenuator.

[c16] 16. An instrument according to Claim 15 comprising a single mode cavity.

[c17] 17. An instrument according to Claim 15 wherein said microwave source is selected from the group consisting of magnetrons, klystrons, and solid state devices.

[c18] 18. An instrument according to Claim 15 wherein said mouth of said reaction vessel is outside of said cavity and outside of said attenuator..

[c19] 19. An instrument according to Claim 15 comprising a waveguide between said source and said cavity.

[c20] 20. An instrument according to Claim 15 further comprising means for venting said vessel without opening said valve to said tube.

- [c21] 21. An instrument according to Claim 20 wherein said valve includes said venting means.
- [c22] 22. An instrument according to Claim 15 further comprising:
a source array; and
a robotic sampler in fluid communication with said source array and with said tube for transferring fluid from said source array to said reaction vessel independent of any movement of said reaction vessel or of said source array or of any source vessels in said source array.
- [c23] 23. An instrument according to Claim 22 further comprising:
a product array; and
a robotic sampler in fluid communication with said product array and with said tube for transferring fluid from said reaction vessel to said product array independent of any movement of said reaction vessel or of said product array or of any product vessels in said product array.